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IS 1448-65 (1986): Methods of test for petroleum and its products, Part 65: Oxidation test for lubricating oils [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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“Knowledge is such a treasure which cannot be stolen”

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IS : 1448 [P : 65] - 1986

(Reaffirmed 2003)

Indian Standard
METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS
[P : 65]
OXIDATION TEST FOR LUBRICATING OILS
(Second Revision)

First Reprint DECEMBER 1998

UDC 665.6/.7 : 621.892 : 543.872

Adapted with permission from the Institute of Petroleum Standard No. IP 48/80 (Reapproved 1986)

1. SCOPE

1.1 This method indicates the tendency of a lubricating oil to deteriorate on oxidation under specified conditions. A measure of the deterioration is obtained by comparison of the viscosity and carbon residue before and after oxidation.

1.1.1 This test is not suitable for additive-type oils (other than those containing ashless additives) or those which form solid products or lose more than 10 percent by evaporation during the test.

2. OUTLINE OF THE METHOD

2.1 The sample is subjected to relatively severe oxidation conditions by heating to 200°C and passing air through it at a rate of 15 litres per hour for two periods of 6 hours each. After standing for 15 to 30 hours, the kinematic viscosity and Ramsbottom carbon residue of the oxidized oil are determined and compared with those from corresponding tests on the unoxidized oil (*see* Note).

NOTE — The above test conditions were designed for laboratories where shift facilities are not available. The full test period is 3 days and this may cause difficulties in refineries where production and laboratory facilities are continuous. In these circumstances, where the test is being used for production control, it is permissible to use a shortened version with a 12-hour continuous oxidation period followed by immediate determination of the viscosity and Ramsbottom carbon residue of the oxidized oil. Practical experience has shown that results obtained by the abbreviated version are normally within the repeatability and reproducibility of the full test procedure.

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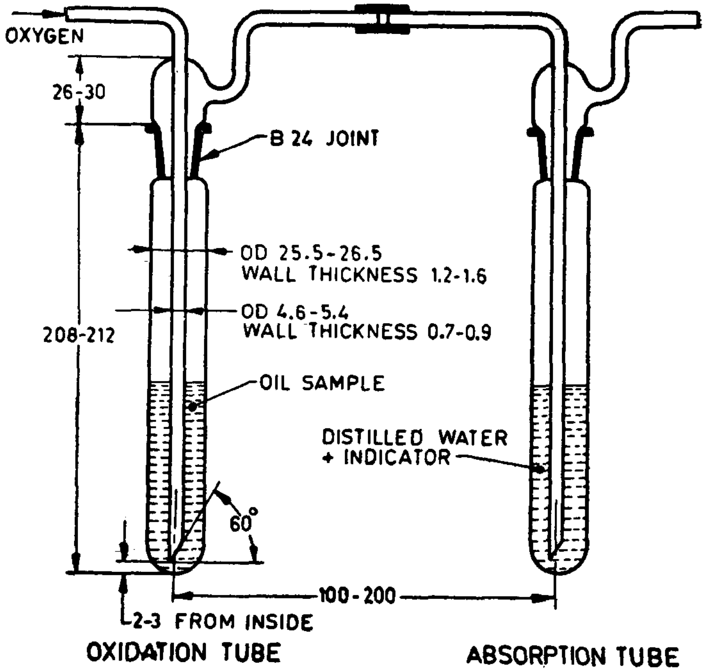
2.2 It is, however, necessary for the test to be carried out in full where specification figures are being verified, or in cases of dispute.

3. APPARATUS

3.1 Oxidation Tubes — Manufactured from brosilicate or neutral glass having the dimensions shown in Fig. 1.



All dimensions in millimetres.

FIG. 1 OXIDATION AND ABSORPTION TUBE

3.2 Absorption Tubes — These are the same as the oxidation tubes. The connection between oxidation and absorption tubes shall be as short as possible and made of glass tubing butt-jointed to the vessels by means of short flexible sleeves* (length between axis of the two tubes 150 ± 50 mm). These tubes are mounted outside the bath.

3.2.1 *Optional Additional Equipment* — Reduction of objectionable atmospheric odour. The oxidation tube used in this method vents to the atmosphere and results in an unpleasant smell in normal laboratories.

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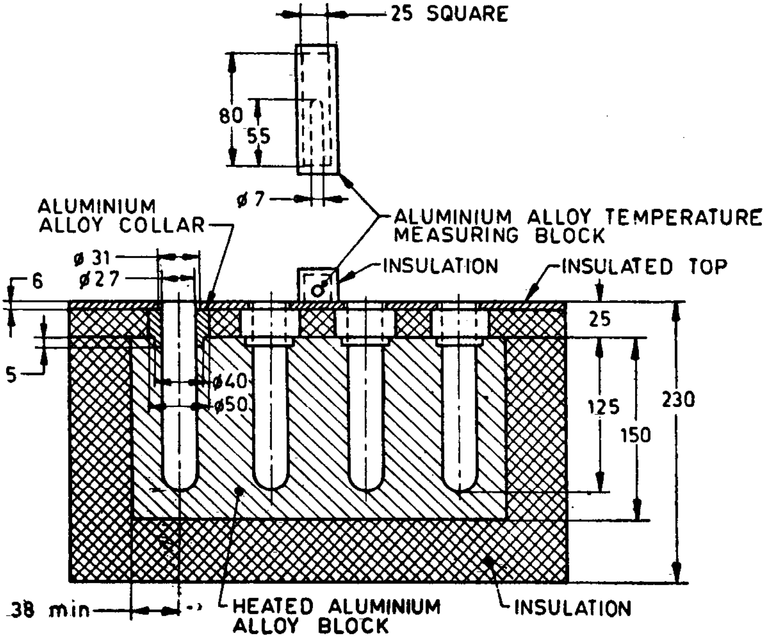
*Silicone rubber sleeving has been found suitable for this purpose.

This can be avoided by passing the issuing vapours through a tube containing water.

3.3 Heating Bath — An aluminium alloy block heater or oil bath thermostatically controlled at a temperature of 200 ± 0·5°C (*see* Fig. 2), the temperature being read from a thermometer (*see* **3.5**) inserted to within 5 mm of the bottom of an oxidation tube containing 40 ml of oil (without air supply). When using an aluminium block heater, the tubes shall be inserted into the walls to an overall depth of 150 mm of which 140 mm shall be heated. In the case of oil baths, the tubes shall be immersed to 140 mm and to an overall depth of 155 mm in the bath. Suitable heat resistant material such as a silicone rubber O-ring can be fitted round the oxidation tube at the level of the top of the heating block to ensure a firm fit.



All dimensions in millimetres.

FIG. 2 TYPICAL METAL HEATING BATH

3.4 Air Supply — Air from a suitable source shall be passed through a cotton wool or glass wool filter into a pressure stabilizer and then to the desired number of oxidation tubes through any system capable of delivering 15 ± 0·25 l/h to each oxidation tube, the rate being measured by any suitable method such as a previously calibrated liquid flow meter.

However, the flow rate shall be checked periodically by the use of a soap bubble flow meter connected to the exit of the oxidation tube or absorption tube, if being used.

3.5 Thermometer — The thermometer* shall comply with the following requirements:

Range	195 to 205°C
Graduation at each	0·1°C
Immersion	100 mm
Overall length	300 ± 10 mm
Stem diameter	5·0 to 8·0 mm
Bulb shape	Cylindrical
Bulb length	25 to 35 mm
Bulb diameter	Not greater than stem
Length of graduated portion	70 to 110 mm
Distance, bottom of bulb to 195°C graduation mark, *Min*	165 mm
Longer lines at each	0·5°C
Figured at each	1°C
Expansion chamber	Required
Top finish	Ring
Scale error not to exceed	± 0·2°C

3.6 Soap Bubble Flow Meter — Suitable for the required flow rate (150 ml capacity) (*see* Fig. 3).

4. PREPARATION OF APPARATUS

4.1 Cleaning the Test Tubes — The oxidation and absorption tubes shall be chemically cleaned. A satisfactory method is to wash with chloroform or petroleum spirit, dry by blowing and then soak with concentrated sulphuric acid for a minimum of 16 h. Drain and completely remove sulphuric acid by washing first with tap water, then with distilled water. Dry the tubes in an air oven at 105 to 110°C for at least 3 h, and then allow to cool to room temperature in a desiccator in which they are kept until use.

5. PROCEDURE

5.1 Transfer 40 ml of the sample at room temperature into the oxidation tube, insert the ground-glass head and place the tube in the heater at

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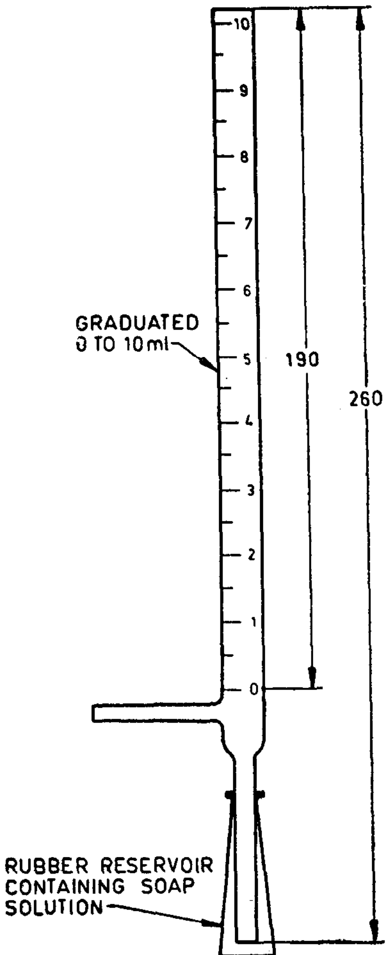
*Thermometer with the Institute of Petroleum designation IP 22 C conforms to these requirements.

200 ± 0·5°C (*see* Note under **5.1.1**). If the absorption tube is being used, add 25 ml of distilled water and connect to the oxidation tube.



All dimensions in millimetres.

FIG. 3 SOAP BUBBLE FLOW METER

5.1.1 To avoid evaporation of the water the absorption tube shall be protected from the bath by insulation.

NOTE — If a number of tubes are to be placed simultaneously in the bath, then the tubes may be preheated for 5 to 10 minutes at 180 to 200°C in order to reduce the cooling effect on the oxidation bath.

5.2 After the oxidation bath has again reached 200 ± 0·5°C and not less than 10 minutes after introducing the oxidation tube, adjust the air flow to 15 ± 0·25 l/h for a period of 6 hours. At the end of this period remove the oxidation tube from the bath and allow it to stand at ambient temperature for 12 to 18 hours.

5.3 Repeat the heating procedure for a further 6 hours and at the end of this time remove the oxidation tube from the bath and allow to cool to ambient temperature.

5.4 Between 15 and 30 hours after completion of the oxidation periods heat the oxidation tube in a boiling water bath and mix thoroughly to ensure the uniformity of the contents.

5.5 Immediately after heating determine the kinematic viscosity at 40°C of the oxidized oil by the method prescribed in IS : 1448 [P : 25]-1976*. If after filtering through a No. 200 (75 μm) sieve as described in the method prescribed in IS : 1448 [P : 25]-1976* successive determinations do not give consistent results, the oil shall be reported as being unsuitable for this method. Using the same instrument, if practicable, determine the kinematic viscosity at 40°C of the unoxidized oil.

5.6 Determine the Ramsbottom carbon residue by the method prescribed in IS : 1448 [P : 8]-1967* of the unoxidized and oxidized oil.

6. CALCULATION AND REPORTING

6.1 The following data shall be reported:

a) Kinematic viscosity of the unoxidized oil at 40°C,

b) Kinematic viscosity of the oxidized oil at 40°C,

c) Viscosity ratio $= \dfrac{\text{Viscosity after oxidation}}{\text{Viscosity before oxidation}}$

d) Ramsbottom carbon residue of the unoxidized oil,

e) Ramsbottom carbon residue of the oxidized oil, and

f) Increase in Ramsbottom carbon residue after oxidation.

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*Methods of test for petroleum and its products :

P : 25 Determination of kinematic and dynamic viscosity (*first revision*).

P : 8 Carbon residue by Ramsbottom method (*first revision*).

7. PRECISION

7.1 Results of duplicate tests should not differ by more than the amounts given in **7.1.1** and **7.1.2**.

7.1.1 *Viscosity Ratio*

Level of Result X	*Repeatability*	*Reproducibility*
1·08 to 2·4	$0·288\,3\,(x - 1·0)$	$0·575\,9\,(x - 1·0)$

7.1.2 *Increase in Ramsbottom Carbon Residue*

Level of Result X	*Repeatability*	*Reproducibility*
0·2 to 2·4	$0·125\,8\,(x + 0·38)$	$0·339\,2\,(x + 0·38)$

where X is the average of two results.

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